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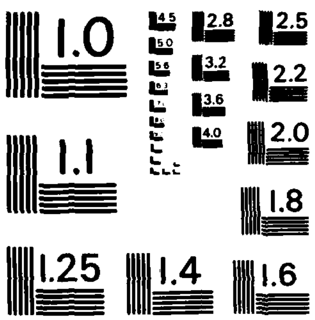

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* Presented at the International Symposium on Protection Against Chemical Warfare Agents, Stockholm, Sweden, June 6-9, 1983.

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ABSTRACT

Paper containing activated charcoal has been produced by dispersing finely-ground charcoal in water along with dispersing agents and adding the mixture to the paper stock before forming the paper. This paper has been produced both as handsheets and on a pilot-scale paper machine.

Optimization of properties such as strength, retention of charcoal in the paper and adsorption capacity is being studied. Charcoal paper could be used as curtains in a contamination control area (CCA) for scavenging chemical agent vapours or as a disposable floor covering in a CCA to reduce the liquid hazard by adsorbing liquid agents transferred to it.

This report outlines the work that has been performed and future plans for the development of charcoal paper.

RÉSUMÉ

On a obtenu du papier contenant du charbon actif en dispersant du charbon réduit en poudre et en versant des agents de dispersion dans de l'eau, ce qui a formé un mélange qu'on a ajouté à de la pâte à papier avant d'en former des feuilles. On a ensuite produit des échantillons d'essai et du papier au moyen d'une machine à papier pilote.

On étudie l'optimalisation de propriétés comme la résistance du papier, sa capacité d'adsorption et de rétention du charbon. Ce papier pourrait servir d'écrans dans une salle de contrôle de contamination pour le balayage des vapeurs d'agents chimiques, ou encore de revêtement de sol jetable pour y réduire les risques que présentent les agents liquides adsorbants qui pourraient y être versés.

Dans le rapport, on donne un aperçu du travail déjà accompli et des projets envisagés concernant la mise au point du papier contenant du charbon.

INTRODUCTION

Activated charcoal is an important chemical protection material. Its ability to adsorb chemical agents, thereby reducing and often eliminating the hazard, is used in several applications including gas mask canisters and protective clothing.

A paper containing activated charcoal has been developed at DREO. There are many possible applications for activated charcoal paper. In a contamination control area, for example, which separates the chemical environment from a clean, agent-free, collective protection shelter, the charcoal paper could be hung like curtains to scavenge chemical vapours. It could also be put on the floor to soak up liquid agent that might fall from or be transferred by contact with clothing, masks, boots or other equipment. The paper could also be used as a disposable adsorbent cape for servicemen during chemical agent attacks.

EXPERIMENTAL

The charcoal paper was made by first dispersing finely-ground activated-charcoal in water with appropriate dispersing agents. This mixture was added to the paper stock before the paper was formed. For most of the experiments, the paper was made by hand but several trials were also performed on the DREO pilot-scale paper machine (1). Whether the paper was made by hand or on the machine, the general principles of paper formation were the same. It was formed by allowing the stock mixture, containing pulp, charcoal particles and added chemicals, to drain through a screen. The pulp and charcoal were retained on the screen while the water flowed through. The paper sheet was then pressed and dried.

It is important that as much as possible of the charcoal be retained in the paper and that the paper be as strong as possible. Many factors affect these two properties. A list of those studied is shown in Table I.

TABLE I.

Factors Affecting Charcoal Retention and Paper Strength

(1) Degree of Beating of Pulp
(2) Nature of Pulp Mix
(3) Charcoal Loading
(4) Charcoal Particle Size
(5) Chemical Additives

(a) Alum - Aluminum Sulphate
(b) Wet and Dry Strength Resins
(c) Dispersing Agents
(d) Retention Aids
(e) Sizing Agents

The first factor was the degree of beating of the pulp. The pulp was beaten by passing a water suspension of the pulp fibres through a narrow gap between a stationary plate and a revolving rotor. The mechanical action on the pulp fibres has several effects including cutting and fibrillation or splitting of the fibres. The nature of the pulp mix refers to the type of pulp that was used. We chose to evaluate three pulps. Hardwood made by the sulphate process and softwood pulp made by both the sulphate and sulphite processes. The charcoal loading or percentage of charcoal in the paper was varied to study the effect on the paper properties. Charcoal particle size was varied from 1.5 to 20 μm. Each of the chemical additives listed was evaluated to discover its effect on the paper properties.

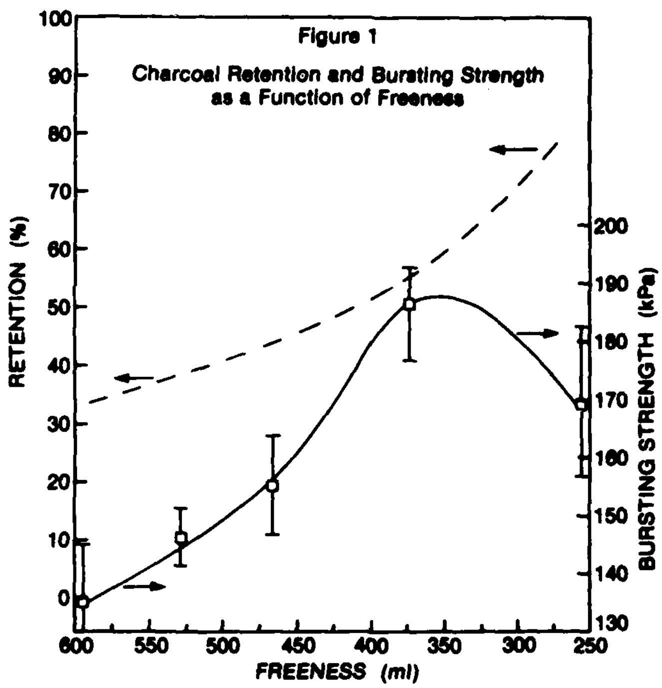
RESULTS AND DISCUSSION

Beating of the pulp had a significant effect on both the retention of charcoal and the strength of the paper (Figure 1). The degree of beating is measured by determining the freeness of the pulp. Freeness

is a measure of how easily water drains away from the fibres while they are being formed into a wet mat on a drainage plate (2). The longer a pulp is beaten, the slower the drainage rate and the lower the freeness value. Beating the pulp causes cutting and fibrillation or splitting of the fibres. Fibrillation increases the surface available for bonding and therefore increases the strength. Cutting the fibres causes a decrease in strength. it can be seen in Figure 1 that the bursting strength (3) peaks at a freeness of 325-375 ml. There was an increase in retention from 34 to 79% as the pulp was beaten because the shorter fibres form a denser mat and retain more charcoal particles. To obtain both a high bursting strength and high charcoal retention, the pulp should be beaten until the freeness reaches a value between 325 and 375 ml.

Figure 1

Charcoal Retention and Bursting Strength as a Function of Freeness



The second factor studied was the nature of the pulp. There are two main categories of wood, hardwood and softwood. These can be processed into pulp by several methods, two of which are called sulphite and sulphate chemical pulping. Combinations of pulp used included a 50/50 mixture of hardwood sulphate and softwood sulphate pulp, a 50/50 mixture of softwood sulphate and softwood sulphite and 100% softwood sulphate pulp. The bursting strength was much higher for the 100% softwood sulphate pulp (Table II). There was little difference between the three formulas as far as charcoal retention was concerned.

TABLE II

Effect of Nature of Pulp on Charcoal Retention and Bursting Strength

Pulp			Freeness	Retention	Bursting Strength
% Hardwood	% Softwood	% Softwood	(ml)	(%)	(kPa)
50		50	326	51	88
	50	50	340	44	91
		100	337	47	162

Table III shows the effect of charcoal loading on the retention and bursting strength. Note that the basis of charcoal percentage was the weight of dry pulp. Although the retention was better for the paper with 29% charcoal added, it contained only 21 g of charcoal per 100 g of paper, whereas paper with 70% charcoal added and only 36% retention contained 26 g of charcoal per 100 g paper.

TABLE III

Effect of Charcoal Loading on Retention and Bursting Strength

Charcoal Added* (%)	Freeness (ml)	Retention (%)	Bursting Strength (kPa)	Amount of Charcoal in 100 g Paper
29	335	72	172	21
50	317	45	153	23
70	328	36	140	26

* Based on weight of dry pulp.

As expected, the bursting strength decreased as the charcoal loading increased. The charcoal particles interfere with the stronger fibre-fibre bonds causing a decrease in strength. A compromise between retention, bursting strength and actual charcoal in the paper would be the use of 50% charcoal loading. It was also found that at high charcoal loadings, the charcoal particles were not strongly bonded into the matrix and could be easily rubbed out.

Next, a series of handsheet experiments was performed to determine the effect of charcoal particle size on the retention of charcoal (Figure 2). Although the results are scattered, a trend can be seen. The retention increased as the particle size increased. At higher particle sizes, the particles came out of the paper very easily. So, even though a higher retention was achieved during the formation of paper with larger particles, mechanical action would cause a high loss of charcoal particles. The charcoal particles must be held tighter in the paper matrix to be able to use larger particle sizes. This could possibly be achieved by using a resin, however, this additive has both advantages and disadvantages as will be seen later.

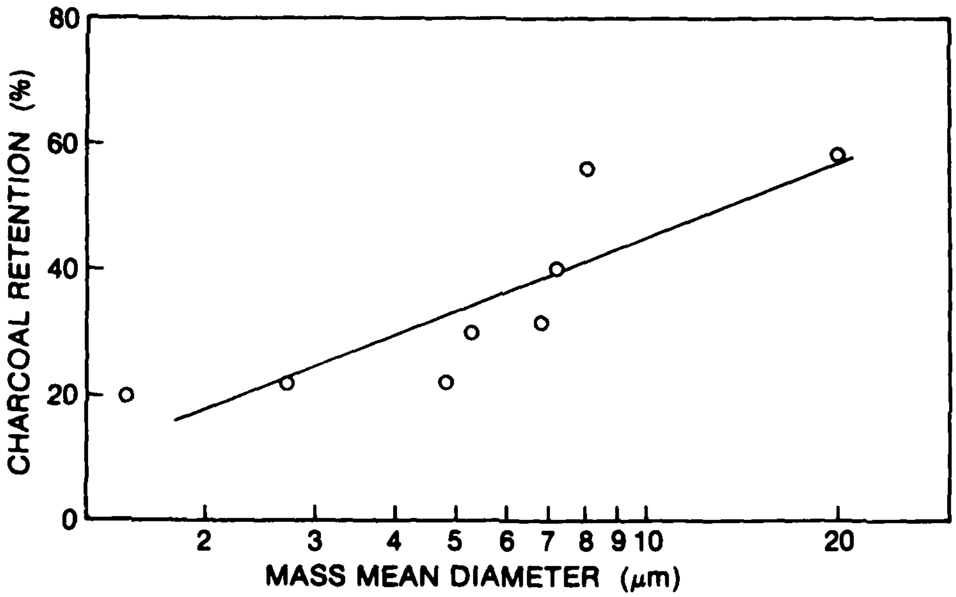


Figure 2: Effect of Particle Size on Charcoal Retention.

The effects of chemical additives on the paper were studied. The use of aluminum sulphate or alum, a common additive in paper making, was investigated first (Figure 3). The experiments showed that the retention increased as the alum content increased. There are, however, two restraints on the use of alum. First, a high alum content can cause

corrosion problems because it is very acidic. Also, it can be seen from the graph that the addition of alum decreased the bursting strength. This decrease is really caused by the increased charcoal content as a result of higher retention. From these results a level of 2-3 percent alum was selected as optimum. It could be possible to work at a higher level of alum, say 4%, if the bursting strength could be increased by the use of an additive such as a resin.

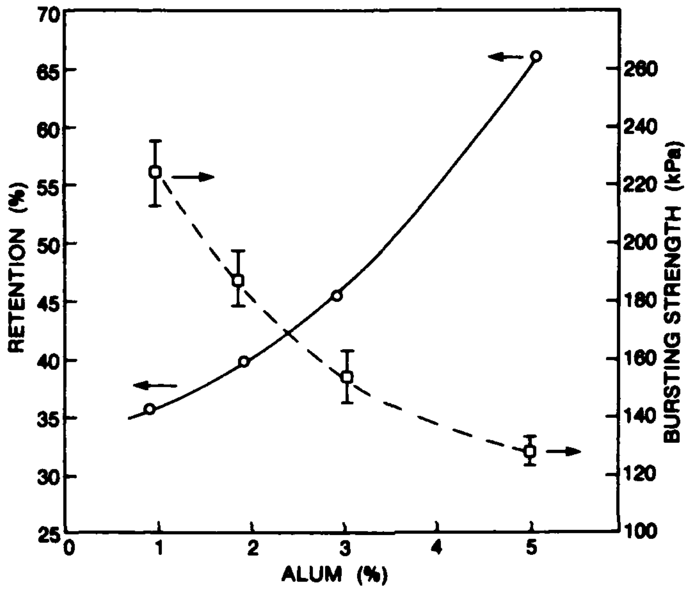


Figure 3: Effect of Alum Content on Charcoal Retention and Bursting Strength.

Table IV shows the effect of various levels of two resins on the retention, bursting strength and the wet-to-dry tensile strength ratio.

TABLE IV

Effect of Resins on Retention, Bursting Strength and Wet/Dry Tensile Strength Ratio

Resin K (%)	Resin P (%)	Freeness (ml)	Charcoal Retention (%)	Bursting Strength (kPa)	Wet/Dry Tensile Strength
-	0	330	59	103	0.10
-	1	330	68	155	0.26
-	3	330	60	154	0.32
-	5	323	56	192	0.40
1.5	-	324	79	133	0.20
3	-	348	76	162	0.30

Resin P which is a melamine-formaldehyde resin was used at levels ranging from 0 to 5 percent. The ratio of wet-to-dry tensile strength (4) increased as the resin content increased. As the level of resin increased, the charcoal retention decreased but the bursting strength increased. Resin K, a polyamide wet-strength resin gave a higher charcoal retention than Resin P at both 1.5 and 3%.

The effect of a retention aid (Table V) on the paper properties was studied. Using retention aid levels from 0 to 0.5% gave little difference in the bursting strength and a slight increase in retention. By comparison, alum at a level of 3% gave a higher retention and lower bursting strength than the paper without alum or retention aid. Finally, incorporation of both alum and retention aid gave a significant increase in the retention of charcoal and a significant decrease in bursting strength.

TABLE V

Effect of Retention Aid on Paper Properties

Alum (%)	Retention Aid (%)	Freeness (ml)	Retention (%)	Bursting Strength (kPa)
0	0	339	42	170
0	0.05	355	42	174
0	0.1	355	43	166
0	0.2	335	45	165
0	0.5	330	48	178
3	0	334	51	141
3	0.2	335	62	117

Before the charcoal particles were added to the pulp, they were mixed with water and dispersing agent. Three dispersing agents were evaluated. A combination of two, one for the actual dispersion and one for the suspension of the charcoal particles in the water gave the best overall performance. The decision to use these two dispersing agents was based on three criteria: the retention of the charcoal, the bursting strength and the uniformity and quality of the surface of the paper. One dispersing agent was rejected because it did not prevent clumps of charcoal from forming on the surface of the paper.

Another additive that can be used in paper making is a sizing agent. Sizing agents are primarily used to give some resistance to aqueous penetration. Table VI shows the effect of varying the sizing agent concentration from 0 to 3 percent. Even accounting for the difference in freeness, both the retention and the bursting strength were higher without any sizing agent. Although the wet-to-dry tensile strength ratio was hard to qualify it was higher with sizing agent than without. It was decided as a result of this data to omit the sizing agent from future formulas.

TABLE VI

Effect of Sizing Agent on Paper Properties

Sizing Agent (%)	Freeness (ml)	Retention (%)	Bursting Strength (kPa)	Wet/Dry Tensile Strength
0.0	324	79	133	0.20
0.5	348	67	122	0.29
1.0	348	65	128	0.23
3.0	343	54	129	0.31

The most promising formulations have been tried on the pilot-scale paper machine. Plans include the continued improvement of the paper to achieve optimum properties for any given application. The paper will also be evaluated to determine how effectively it scavenges chemical agent vapour under various conditions.

Scanning Electron Microscope Photographs

In addition to the experimental testing and evaluation of the charcoal paper, scanning electron microscope photographs were taken to observe the dispersion of the charcoal in the paper.

Figures 4 (04515) and 5 (04521) are the surface of the paper at two magnifications. The charcoal particles are clearly visible and are seen to be uniformly distributed amongst the pulp fibres. The fibres are in the form of long ribbons, as a result of the beating, and are laid down in random direction. The larger charcoal particles are about 5 μm in diameter. In Figure 5, pulp fibrils can be seen attached to the particles.

Figure 6 (04524) is a cross section of the paper showing distribution of the charcoal throughout the thickness of the paper.

ACKNOWLEDGEMENT

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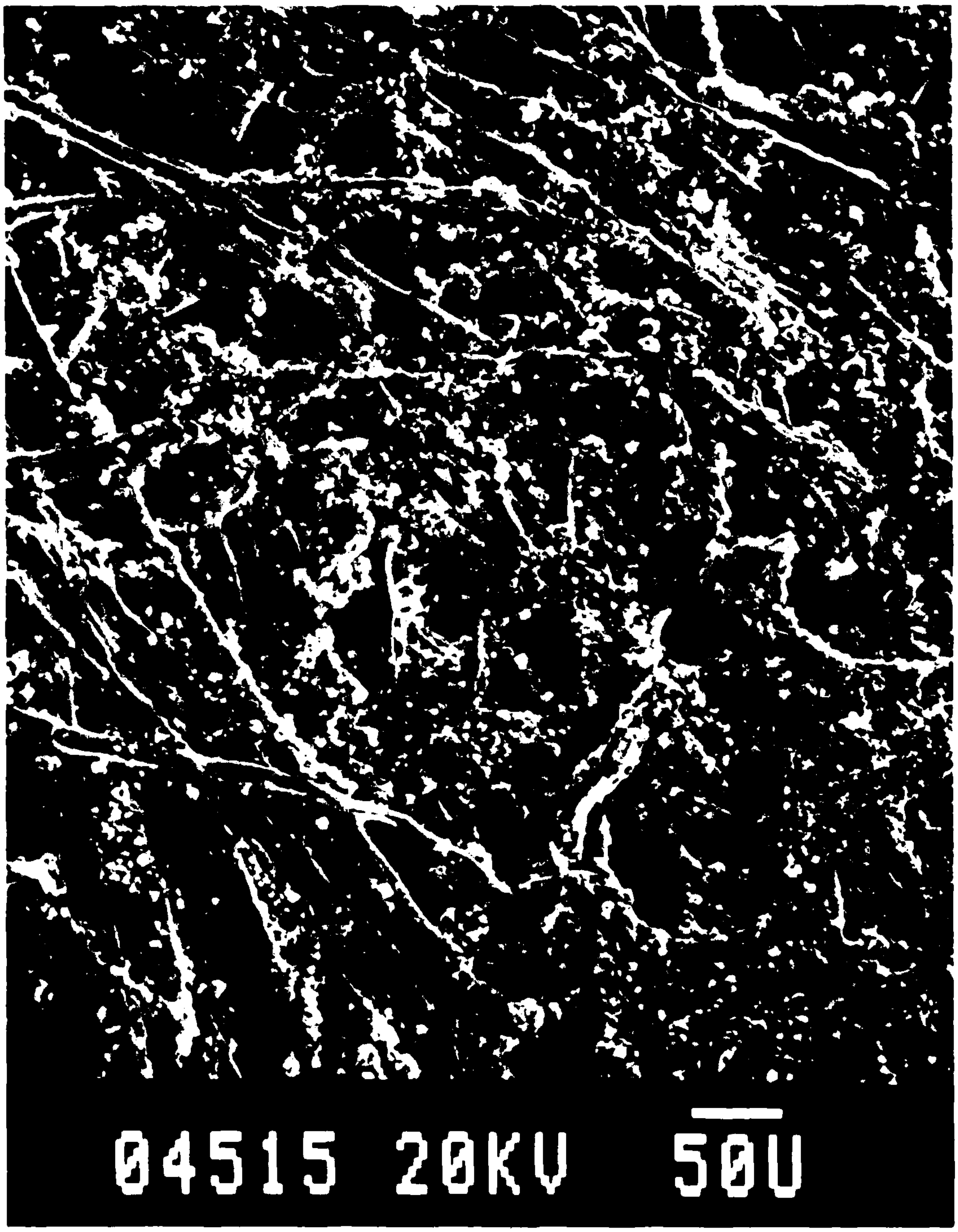

Figure 4: Scanning Electron Microscope Photograph of the Surface of Paper Containing Activated Carbon.

Figure 5: High Magnification of Figure 4.

Figure 6: Scanning Electron Microscope Photograph of a Cross-section of Paper Containing Activated Carbon.

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